Food Hydrocolloids 45 (2015) 186-195

Contents lists available at ScienceDirect

Food Hydrocolloids

journal homepage: www.elsevier.com/locate/foodhyd

The influence of co-solutes on tribology of agar fluid gels

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ARTICLE INFO

Article history: Received 2 September 2014 Accepted 17 November 2014 Available online 25 November 2014

Keywords: Lubrication Tribology Hydrocolloid Fluid gel Agar Rheology

ABSTRACT

The effects of glucose and glycerol on the lubrication properties of agar fluid gels have been studied using soft tribology. A novel approach using the sediment and supernatant of centrifuged fluid gels has allowed investigation of the distinct contributions of both the gelled particulate phase and the continuous phase on fluid gel tribology. The friction coefficient of both the particulate phase and fluid gels was significantly lower than that of the continuous phase across the three lubrication regimes. This indicates that particle entrainment occurs at all entrainment speeds, enhancing lubrication by prevention of surface contact.

Softer fluid gel particles produced with intermediate levels of glycerol (up to 30%) show increased friction as would be expected for an increased contact area between the tribological surfaces. At high levels of glycerol, the friction does not increase. It is proposed that soft particles are produced but the increasing friction is overcome with the increased lubrication from the more highly viscous continuous phase. In contrast, the presence of intermediate levels of glucose (up to 30%) increases the friction of the aqueous continuous phase but does not affect the particle properties. Texture analysis, rheology and light scattering techniques were used to elucidate the structural changes of the fluid gels induced by the addition of co-solutes and the influence this has upon lubrication.

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1. Introduction

Fluid gels are concentrated suspensions of micro-gel particles dispersed in a continuous medium, typically water, and are produced by applying a shear field to the hydrocolloid solution while the sol-gel transition is taking place (Norton, Frith, & Ablett, 2006; Norton, Jarvis, & Foster, 1999). Application of fluid gels is of increasing interest to the food industry because of their ability to generate film thickness in the flow conditions that prevail during food processing in the mouth, potentially imparting desirable mouthfeel and texture attributes (e.g. smoothness) (Malone, Appelqvist, & Norton, 2003). The lubrication properties of fluid gels, which have been shown to be greatly influenced by the physical properties of particles (e.g. size, elasticity) (Gabriele, Spyropoulos, & Norton, 2010; Mills, Koay, & Norton, 2013), can be measured using tribology (thin-film rheology).

In this work, we used fluid gels produced from agar which is a seaweed hydrocolloid composed by mixtures of neutral agarose and charged agaropectin, in proportions that vary depending on both the production process and the raw material (Araki, 1956;

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Phillips and Williams, 2009). Agarose is the polysaccharide with the gelling capability of agar and consists in alternating units of 3linked β -D-galactopyranose and 4-linked 3, 6-anhydro- α -L-galactopyranose (Araki, 1956). The gelling temperature of agar varies depending on the degree of methoxylation of the agarose chain which is characteristic of the seaweed speces from which the agar has been extracted (Armisen & Galatas, 2000).

As the gelling temperature of agar is approached, intramolecular hydrogen bonds start to form between the agarose chains, followed by the formation of inter-molecular hydrogen bonds between the two distinct polysaccharide chains (Tako & Nakamura, 1988). The mechanism for gelation involves a coil to double helix transition and a subsequent aggregation of these helical domains to create a three dimensional structure (Arnott et al., 1974; Morris, 1986; Schafer & Stevens, 1995).

Upon cooling the agar solution under shear below the agar transition temperature, kinetic competition between the process of gel network formation and the shear induced break-up of the network occurs. The result of these two competing mechanisms determines the physical properties of the resulting fluid gel particles as well as the extent of inter-particle interactions. The control over the conformational ordering kinetics is achieved by the applied cooling rate such that a range of viscoelastic properties can be produced by manipulation of the processing conditions.







http://dx.doi.org/10.1016/j.foodhyd.2014.11.014

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The fluid gel formation is kinetically controlled by the number of available junction zones, where the rate of ordering is a function of polymer concentration, temperature and solvent quality. Thus, it is expected that changes in the degree of solvation of the hydrocolloid due to the presence of co-solutes will greatly affect the structure of the fluid gel particles and hence their lubricating properties. Although the effects of sugars and polyols on the material properties of quiescent agar gels have been widely reported in the literature, no published study has investigated the formation and lubrication properties of fluid gels mixed with such co-solutes.

The first objective of this work is to provide a comprehensive understanding of the mechanisms underlying the lubrication properties of agar fluid gels. The lubricating behaviour of agar fluid gel was investigated by Gabriele et al. (2010) who proposed a conceptual model for fluid gel lubrication. However, in order to rationalize these findings in terms of particle properties, it is required to decouple the relevant contributions of both the continuous phase and the particulate phase to fluid gel lubrication. To this end, we have employed a new approach using the supernatant and the sediment of centrifuged fluid gels to evaluate the different contributions.

The second objective of our study is to investigate the effects of co-solutes on fluid gel lubrication by using the proposed approach. In this work, glucose and glycerol were used to modify the solvent quality and the resulting changes in both particle structure and fluid gel properties were measured using tribology, rheology, texture analysis and light scattering techniques. Results show the substantial effect that both the continuous phase and particulate phase have on fluid gel lubrication. The observations from this study also suggest that controlling particle properties allows the lubrication properties to be manipulated, potentially enabling desirable sensory properties to be produced.

2. Materials and methods

2.1. Fluid gel production

Agar (A1296), p-glucose (G7021) and glycerol (49781) were purchased from Sigma Aldrich and used without further purification.

Agar was dispersed in distilled water at room temperature (≈ 20 °C) and then heated while stirring to 90 °C until completely dissolved. The temperature was then lowered to approx. 70 °C and co-solutes were dissolved into the hot solution.

Fluid gels were produced by pumping the hot solutions into a jacketed pin-stirrer device at a constant flow rate of 25 ml/min. The temperature of the cooling unit was kept at 15 °C and the pin-stirrer was operated at a shear rate of 2000 rpm which is the maximum operation speed of the equipment.

Fluid gels with more than 30% glucose could not be produced reproducibly and thus data is not presented.

2.2. Tribology of fluid gels

The friction of the produced fluid gels was measured using a mini traction machine (MTM, PCS Instruments, London).

The tribometer was equipped with an elastomer disc (Samco Silicone Products) and a ³/₄ inch diameter AISI 400 stainless steel ball (PCS Instruments, London) as contact surfaces. These specific materials were selected since they have been previously shown to provide data with correlations to sensory perception (Malone et al., 2003). Fresh silicone discs were used for each test after cleaning by sonication for 5 min in ethanol followed by 5 min in distilled water. The stainless steel ball was cleaned with acetone and distilled water.

A normal load (W) of 2 N and a speed and slide-roll ratio (SRR) of 50% were used for all the experiments as they were found to yield the most reproducible data. Stribeck curves were generated by measuring the friction upon increasing the speed from U = 1-1000 mm/s followed by decreasing the speed from U = 1000-1 mm/s until a total of 6 runs were completed. Each test, consisting in 3 ramps up and 3 ramps down, was repeated 3 times. The error bars represent the 1 standard deviation of the mean of the 18 tests per fluid gel sample.

2.3. Particle size measurements

Particle size distribution was obtained using a Malvern Mastersizer 2000 with a water dispersion unit. A refractive index of 1.42 was used for the agar fluid gel particles, as previously reported by Gabriele et al. (2010).

2.4. Texture analysis

Compression test were performed on quiescently cooled agar gels using a TA.XT.plus Texture Analyser (Stable Micro Systems Ltd., UK) with a 40 mm diameter cylindrical aluminium probe. The height and diameter of the gel cylinders were 10 mm and 23 mm, respectively and the compression rate was 1 mm/s. The true stress σ_T and true strain ε_H were calculated using the following equations, as described elsewhere (Moresi & Bruno, 2007; Norton, Cox, & Spyropoulos, 2011):

$$\sigma_E = F/A_0 \tag{1}$$

$$\varepsilon_E = (h - H_0)/H_0 \tag{2}$$

$$\epsilon_H = \ln(1 + \epsilon_E) \tag{3}$$

$$\sigma_T = \sigma_E (1 + \varepsilon_E) \tag{4}$$

where H_0 and A_0 are the initial height and cross-sectional area, h and F are the height and the compression force applied, ε_E and ε_H are the engineering and true (Henky) strain and σ_E and σ_T are the engineering and true stress.

2.5. Rheological tests

Dynamic oscillatory measurements were performed on a Kinexus Rheometer (Malvern Instruments Ltd., UK) using a cone and plate geometry (4 °cone angle, 40 mm diameter).

Strain and stress sweep tests were carried out at 1 Hz on fluid gels 48 h after production.

3. Results and discussion

3.1. Effect of co-solutes on quiescent gels

Previously reported DSC studies on agar fluid gels produced using the method outlined in this paper showed that the enthalpy of melting of fluid gels is comparable to that of their quiescently cooled counterparts (Norton et al., 1999). This indicates that the intrinsic polymeric network and therefore the textural properties of the agar fluid gel particles are the same as those of the corresponding quiescent gels, as suggested by Frith, Garijo, Foster, and Norton (2002). Thus, in order to elucidate the effects of glycerol and glucose on gel particle stiffness, compression tests on quiescent gels were carried out. Download English Version:

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